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THE EFFECT OF MECHANICAL WORKING ON SIC WHISKER-REINFORCED ALUM--ETC(U)
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THE EFFECT OF MECHANICAL WORKING ON SiC WHISKER-REINFORCED ALUMINUM ALLOYS

DENNIS M. RIGGS and PETER GILLIS
APPLIED SCIENCE DIVISION

April 1980

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ABSTRACT

The effects of mechanical working on SiC whisker-reinforced aluminum alloys were investigated. The effects of pressing, extruding, and rolling on whisker dispersion, L/D ratio, and orientation were determined by examination of polished cross sections using conventional optical microscopy and by examination of replicas with a transmission electron microscope. These results were correlated with the mechanical properties of the composite. It was found that mechanical working of the composites significantly degrades the whiskers, particularly the L/D ratio. Extrusion and rolling, however, do impart an orientation to the whiskers in the direction of working. It was also found that clumping observed in the unworked billets is carried through both the extrusion process and rolling. These SiC-rich areas apparently lead to the formation of porosity in the composites during working. The high level of mechanical properties found in the worked composites suggests that optimization of the secondary working parameters (in such a manner as to minimize whisker damage) may result in the realization of a truly unique structural material.

INTRODUCTION

The development of a low-cost process for producing silicon carbide whiskers from rice hulls has spurred a renewed interest in the development of silicon carbide whisker-reinforced aluminum and aluminum alloys. The reinforcement of aluminum alloys with whiskers has been an area of keen interest for a number of years. This interest has centered on the premise that whisker reinforcement would allow structural designers to utilize a material which is not only strong, stiff, and lightweight, but also capable of being mechanically worked and shaped. This factor of mechanical working has been a major limitation to the widespread usage of continuous filament-reinforced metal matrix composites.

In the past, incorporation of SiC whiskers into aluminum alloys has been a problem, particularly in terms of attaining good wetting, bonding, and dispersion. In 1969, however, Lare and Divecha[1] developed a process in which incorporation of the whiskers into the alloys became much easier. Essentially, they discovered that a good, well-bonded composite could be attained by heating a mixture of SiC whiskers and aluminum alloy powders under pressure to a temperature near the liquidus of the alloy followed by cooling under pressure to below the solidus. Mechanical working of these "as-pressed" billets was found to be fairly easy. The extrusion of bars, tubing, and crosses was demonstrated as well as the rolling of thin sheet, forgings, and so on.

In this report, an electron microscopy study of the effects of mechanical working on the physical characteristics of the whiskers incorporated within the aluminum alloys will be discussed. In particular, emphasis will be placed on damage incurred by the whiskers during working, whisker dispersion, and L/D ratios. As-pressed material, extruded bars, and rolled sheet will be examined.

WHISKER CHARACTERISTICS

The whiskers used to fabricate the composites which were studied in this investigation were produced by Silag, Inc., from rice hulls. Typical properties of these whiskers (Type M-8) are listed in Table 1. As can be seen, the whisker content of M-8 SiC is about 15 to 25% with the remainder being composed of SiC powder. The whiskers tend to have diameters ranging from 0.2 to 0.5 micron and L/D ratios from 20 to 100.

Table 1. CHARACTERISTICS OF SILAG, INC., M-8 SiC*

Whisker Content, %	15 to 25
Particle Content, %	75 to 85
β-SiC, wt %	97 to 98
SiO_2, Si_3N_4, etc., wt %	0 to 0.5
Free Carbon, wt %	1
Other Metallic or Oxide Impurities, wt %	0.5
Particle Size Average, microns	3.2
Density, g/cc	3.2
Tensile Strength (whiskers), psi	0.6 to 3.0×10^6
Tensile Modulus (whiskers), psi	70 to 120×10^6

*Product Specification Sheet, Silag, Inc.

1. LARE, P. J., and DIVECHA, A. P. *Consolidation and Extrusion of Fiber-Reinforced Composites.* U.S. Patent 3,833,697, Melpar, Inc., September 1974.

COMPOSITE FABRICATION AND PROPERTIES

Three types of composite products have been examined: extruded bar, rolled sheet, and as-pressed material. The extruded bar and rolled sheet were fabricated at the Naval Surface Weapons Center while the as-pressed material was obtained from DWA Composite Specialties.

Billets for the extruded bar and rolled sheet were fabricated as generally outlined in NSWC/WOL TR 78-142.[2] Essentially, a blended mixture of 25 v/o SiC and 2024 aluminum alloy powder was cold compacted in a graphite/steel die assembly. The die, containing the blended mixture, was heated to a temperature near or above the liquidus temperature of the matrix alloy while a pressure of about 5000 psi was maintained. The die assembly was then cooled to a temperature below the solidus of the matrix while maintaining this pressure. After cooling, the composite billet was ejected and shaped into its final configuration. The extruded bars used in this study were hot extruded, a ratio of 10.5:1 to a final diameter of 0.375 inch. The rolled sheet was produced by rolling an extruded bar in a direction normal to the extrusion direction. The rolling was accomplished at 840 F at 0.0025 inch per pass. The final sheet thickness was 0.020 inch.

The as-pressed material used in this study consisted of SiC whiskers in 7075 aluminum. The material was produced using a proprietary process by DWA.* In general, a mechanically mixed blend of M-8 SiC, aluminum alloy powder, and a fugitive carrier was placed in a heated tool, evacuated, and subsequently pressed and sintered. The resulting panels were then removed and cleaned up.

Mechanical property measurements were made on the extruded bars and the as-pressed material. Due to a lack of sufficient material, no mechanical properties were measured for the rolled sheet. Modulus values for the extruded bars ranged from 18 to 25×10^6 psi. Strength measurements as a function of temperature, obtained at AMMRC, are shown plotted in Figure 1. The bars used in obtaining the

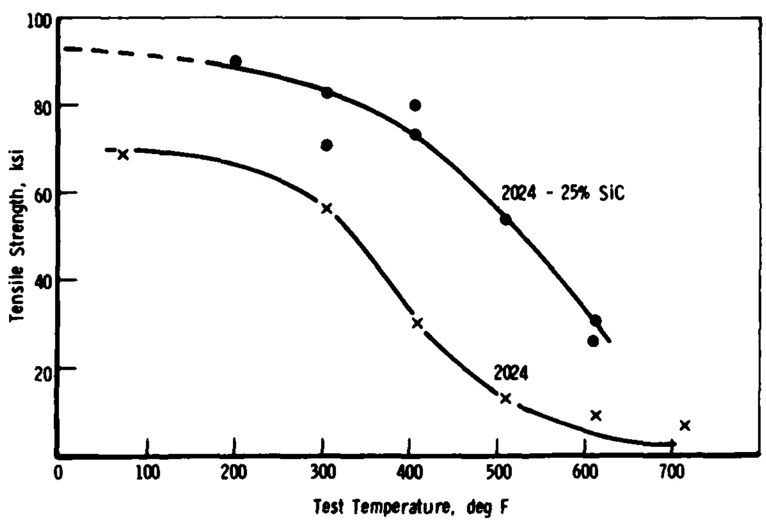


Figure 1. Ultimate tensile strength of SiC whisker-reinforced 2024 aluminum as a function of temperature.

*Personal communication, J. Dolowy, DWA Composite Specialties.

2. DIVECHA, A. P. et al. *Progress in Preparation and Deformation of Silicon Carbide-Aluminum Metal Matrix Composites.* NSWC/WOL TR 78-142, September 1978.

values of tensile strength were initially in the T-4 condition. The specimens were heated to test temperature and held 15 to 20 minutes before being loaded. As can be seen in the figure, the ultimate tensile strength of the composite is significantly greater than that of unreinforced 2024 at any given temperature. Room temperature tensile strengths of about 90,000 psi have been observed compared to values of 68,000 psi for the unreinforced material. The ultimate strength remains fairly constant to about 400 F. Yield strengths at 0.2% offset are also quite high for the 25 v/o SiC-reinforced 2024. Values greater than 85,000 psi were measured at 200 F. At 500 F, the yield strength averaged 47,000 psi.

The as-pressed material was tested by DWA. The composite contained 20 v/o SiC in 7075 aluminum alloy. Its room temperature ultimate strength was 56,200 psi and its modulus was 16.1×10^6 psi.

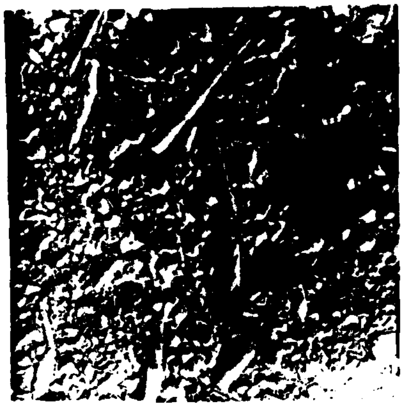
CHARACTERIZATION OF AS-PRESSED SiC/7075 ALUMINUM

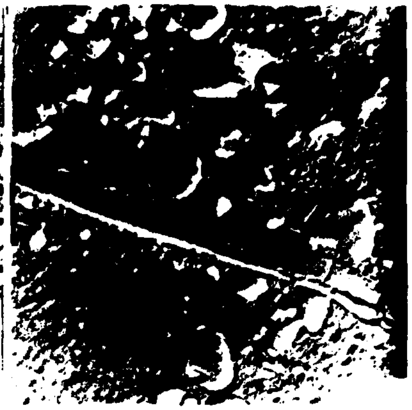
The as-pressed material of 20 v/o SiC in a matrix of 7075 aluminum was examined by pulling replicas from polished and lightly etched sections parallel to the surface and from the cross section of the as-pressed plate. The replicas were observed in an electron microscope. The results of this examination are shown in Figure 2.

Figures 2a-d are from polished sections parallel to the surface of the plate. As can be seen from Figure 2a, a considerable amount of SiC powder and a lesser amount of SiC whiskers are readily apparent. The majority of the powder is undoubtedly from the high powder content ($\sim$75%) of the M-8 whisker product. The average L/D ratio of the whisker material which is visible would appear to be fairly high but an accurate number cannot be obtained from this micrograph. Figure 2b shows another area from the same specimen; this micrograph shows a very long, thin whisker with a screw-like appearance and immediately adjacent to the whisker, an area which appears to be an inclusion. This apparent inclusion is not thought to be an artifact of the replication process. In addition, this region appears to be somewhat depleted in SiC compared to the area shown in Figure 2a. Figure 2c shows an area where some whisker damage has occurred. The large whisker in the center of the micrograph exhibits a distinct split, particularly near the whisker end. The split may be the result of the polishing process but this cannot be confirmed. A number of hexagonal crystals, which are probably whisker ends oriented perpendicular to the plane of the photograph, are also visible. Figure 2d depicts another area of the composite where clumping of the SiC powder and whiskers is evident.

Figures 2e-h were obtained from the polished cross section of the as-pressed plate. Figure 2e shows evidence that clumping of SiC powder and whiskers occurs during processing. In addition, the processing also seems to have resulted in considerable damage. Extensive breakup is also seen in Figure 2f. Breakup of the whiskers has occurred both along the length of the whisker and across the diameters. Figure 2g shows a mixture of powder, hexagonal whisker ends, and slightly damaged whiskers. Figure 2h shows an area of sparse SiC content.

The results of this examination indicate that the whiskers and SiC powder are apparently well bonded to the 7075 aluminum alloy matrix, but clumping and areas of extensive whisker damage are present. In those areas where whisker damage has





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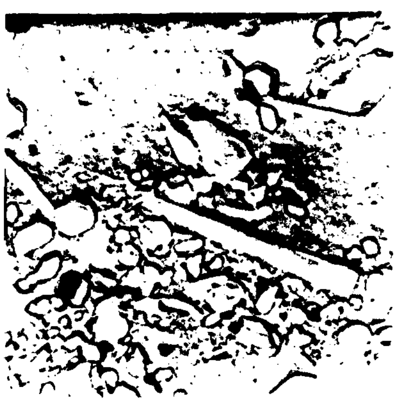
(d) Mag. 9600X (e) Mag. 5100X (f) Mag. 5100X



(g) Mag. 9600X (h) Mag. 9600X

Figure 2. Replicas of as-pressed SiC/7075 aluminum examined in a transmission electron microscope. Figures a-d are sections parallel to the face of the as-pressed plate. Figures e-h are sections perpendicular to the face of the plate.

occurred, the average L/D ratio of the whiskers is rather small. In other areas, where damage has not been so extensive, the L/D appears usually to have a value of at least 10 or more. Distinct differences also seem to exist among the whiskers themselves, particularly in their diameters. Some of the whiskers have rather large diameters compared to others and a number of the whiskers have hexagonal cross sections. The hexagonal cross section may be due to a growth rate effect for β-SiC or it may indicate the presence of α-SiC.

CHARACTERIZATION OF EXTRUDED SiC/2024 ALUMINUM

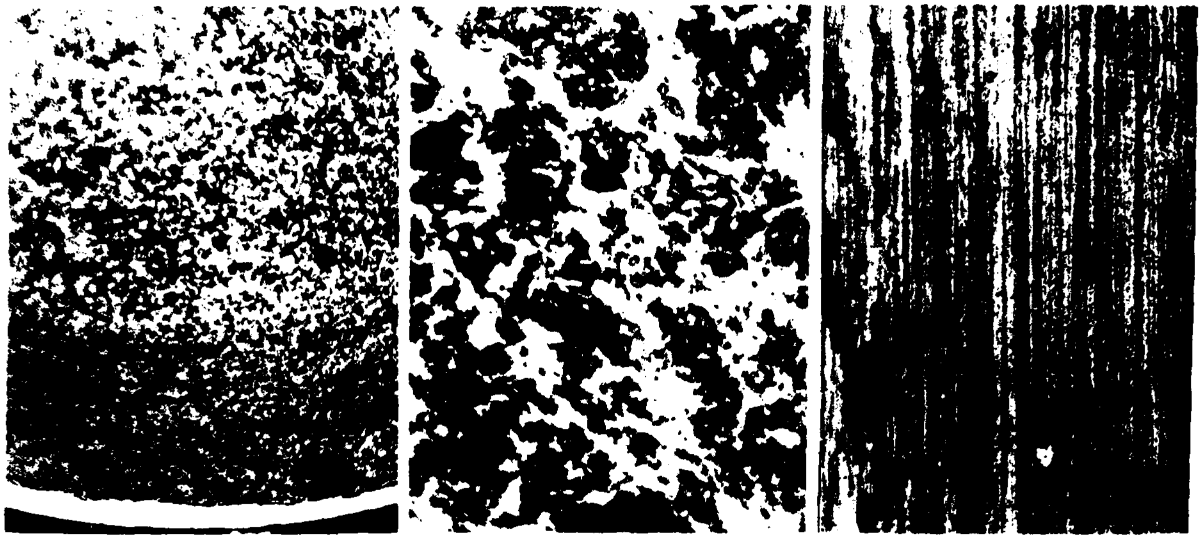
General

The extruded bars of 25 v/o SiC whiskers in a matrix of 2024 aluminum were examined by conventional optical microscopy and by examining replicas of polished and lightly etched specimens with the electron microscope.

Optical Microscopy Observations

The results of the optical microscopy study are shown in Figure 3. Figures 3a and 3b were obtained from polished cross sections of the bar while Figures 3c-e are from longitudinal sections. As can be seen from Figure 3a, a distinct difference exists between the center of the extruded bar and regions near the surface. The surface region, for example, possesses a considerably higher content of grayish material, suggesting that a significantly higher concentration of SiC exists near the surface as compared to areas near the center of the bar. A distinct boundary between the surface region and the center of the bar is apparent. In addition, Figure 3a also shows that the surface region exhibits considerable porosity. In Figure 3b is shown a higher magnification view of the center of the cross section of the bar. This micrograph shows that the SiC material is definitely clumped together. Areas with no whisker material are immediately adjacent to areas of very high SiC content. In Figure 3c is shown a longitudinal section from the same bar. This micrograph shows, as did that in Figure 3a, that a higher concentration of SiC is found near the surface of the bar while the center of the bar is apparently depleted. In addition, it can also be seen that the extrusion process had resulted in the formation of numerous SiC-rich bands oriented parallel to the extrusion direction. The width of these bands varies from the center of the bar to the surface with the coarser ones being located near the center. Between neighboring bands are matrix-rich areas with relatively few SiC whiskers. This can be seen a little more clearly by close examination of the micrograph in Figure 3d, where very few whiskers can be seen in the white areas among the bands while within each band arrays of whiskers generally oriented in the direction of extrusion are plainly visible. These results indicate that the "clumped" areas in Figure 3b are actually "stringer" ends in the plane of the cross section. The stringers apparently result from extruding as-pressed materials containing inhomogeneous dispersions (or clumps).

The effects of SiC clumping and stringer formation are probably quite detrimental with regard to the mechanical properties of the composite in that stress concentrators can arise as well as areas with little or no infiltration, thereby inhibiting stress transfer mechanisms from the matrix into the whiskers. Whisker clumping also appears to be detrimental during extrusion as can be seen by examination of Figures 3c and 3e. Considerable porosity, as well as some inclusions, are immediately apparent. Most of the porosity seems to be associated with the SiC-rich stringers and this is probably due to the reasons cited above.



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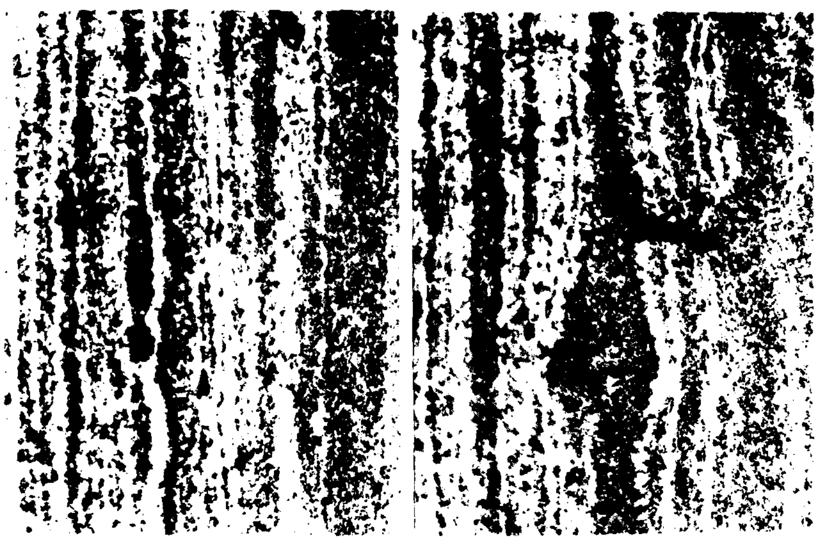


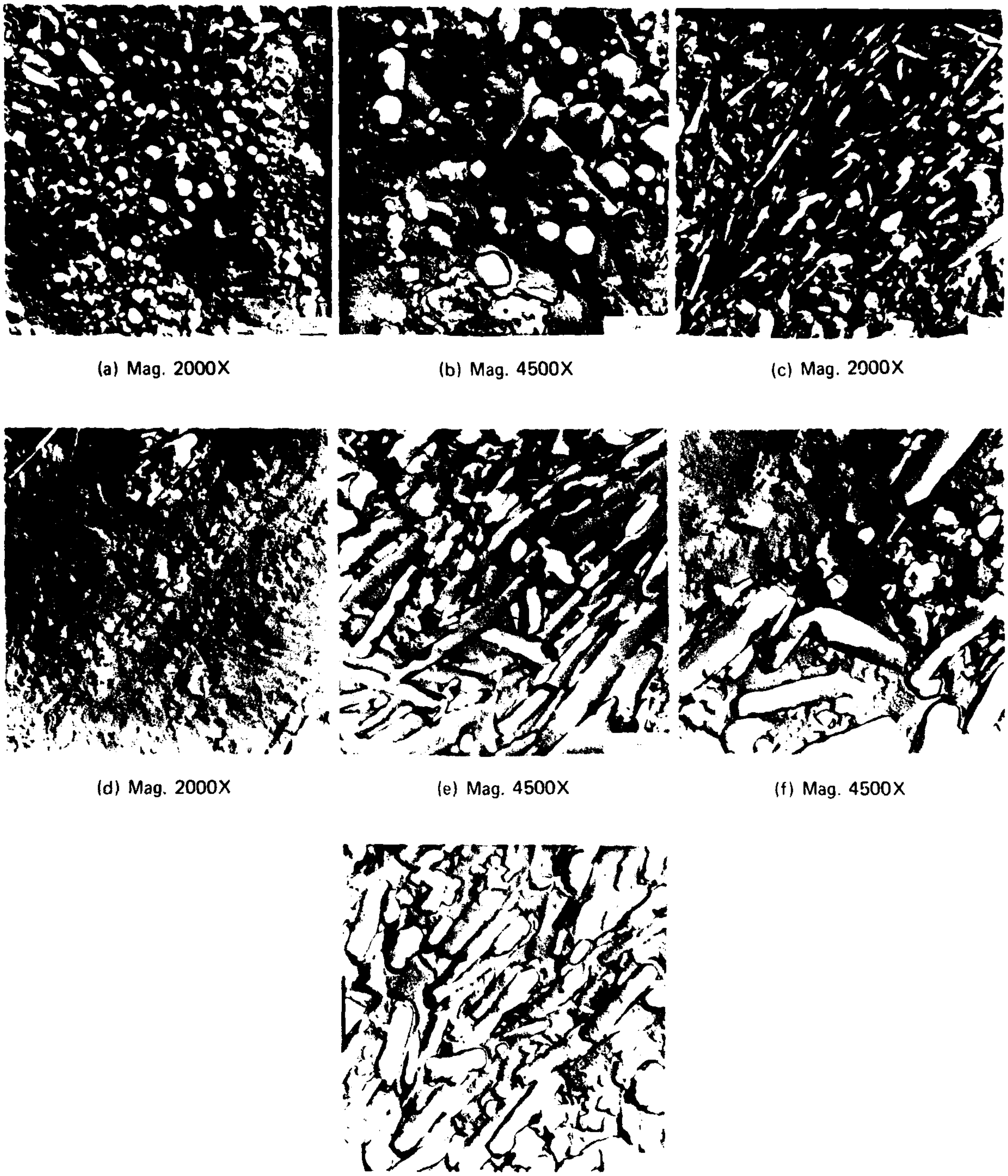
(d) Mag. 225X (e) Mag. 225X

Figure 3. Optical micrographs of polished and etched specimens of extruded 2024 aluminum with 25 v/o SiC whiskers. Figures a and b are cross sections and Figures c-e are longitudinal sections.

Electron Microscopy Observations

Replicas of the extruded bar were made and examined in an electron microscope. The results of those examinations are shown in Figure 4.

Figure 4a is a cross-sectional view and shows considerable clumping of the SiC whiskers and powders. Areas completely void of whiskers and powder are surrounded by other areas of very high SiC content. In many cases, as shown in Figure 4b, intimate contact occurs between neighboring SiC particles, thereby inhibiting stress transfer mechanisms.



(a) Mag. 2000X (b) Mag. 4500X (c) Mag. 2000X

(d) Mag. 2000X (e) Mag. 4500X (f) Mag. 4500X

(g) Mag. 4500X

Figure 4. Replicas of extruded 2024 aluminum with 25 v/o SiC. Extrusion ratio was 10:1.

In Figures 4c and 4d are shown longitudinal sections of the extruded bar. Figure 4c shows that the extrusion process has resulted in a fairly good general orientation of the whiskers in a direction parallel to the extrusion direction. This orientation was not accomplished without causing damage to the whiskers, however. The average L/D for whiskers shown in this and subsequent micrographs was about 6. In addition, the extrusion process has also resulted in numerous whisker-whisker contacts with possibly little or no matrix material in between. Evidence that clumping of the whiskers has occurred is seen by comparing Figures 4c and 4d. Figure 4d is an area which exhibits almost no SiC material at all. Finally, shown in Figures 4e, 4f, and 4g are longitudinal sections of the bar. These three micrographs again show that the extrusion process has resulted in a preferred alignment of the whiskers as well as a general breakup of the whiskers.

Summary

The combination of optical microscopy and electron microscopy for characterizing extruded SiC whisker/2024 aluminum composites has shown that considerable damage can be imparted to the whiskers as a result of the extrusion process. In addition, inhomogeneities in the dispersion of the whiskers in the as-pressed material are carried through the extrusion process and result in the formation of long stringers of SiC material in the extruded bar. Within these stringers, the whiskers and SiC powder come into intimate contact and probably reduce the overall capacity of the matrix alloy to transfer stress into the whisker under loading conditions. In addition, the intimate whisker-whisker contact and clumping effects cause porosity to form during the extrusion process. This porosity is most likely detrimental and probably will continue to occur as long as major inhomogeneities exist in the whisker dispersion.

CHARACTERIZATION OF ROLLED SiC WHISKERS/2024 ALUMINUM

The final part of this study consisted of an electron microscopy examination of longitudinal and transverse specimens from a 0.020"-thick rolled sheet of SiC whisker-reinforced 2024 aluminum. As mentioned in a previous section, the sheet was rolled at 840 F at 0.0025 inch per pass. The material which was rolled was originally an extruded bar having a thickness of 0.375 inch. The bar was rolled in a direction normal to the extrusion direction.

The results of the microscopic examination are shown in Figure 5. Figures 5a-d are longitudinal sections of the sheet. As can be seen in Figure 5a, clumping of the SiC material and extensive breakup of the whiskers has occurred. Areas of little or no whisker content are plainly visible. These same conclusions can be drawn about the area shown in Figure 5b. Figure 5b is interesting in that a number of the hexagonal-shaped crystals discussed previously have become more rounded and appear to be breaking into smaller pieces. It is unclear whether these hexagonal crystals are whisker ends or powders. The rounding of the whiskers is probably a grinding effect resulting from the rolling powder. The conclusion, however, is that the rolling process has imparted considerable damage into the SiC material. This can be seen even more vividly in Figure 5c, where an area containing a number of extensively damaged whiskers is shown. The rolling process has broken the whiskers in a rather periodic fashion into pieces having L/D ratios of about 2. In addition to the breakup of the whiskers this micrograph is also notable because of the absence of other SiC material aside

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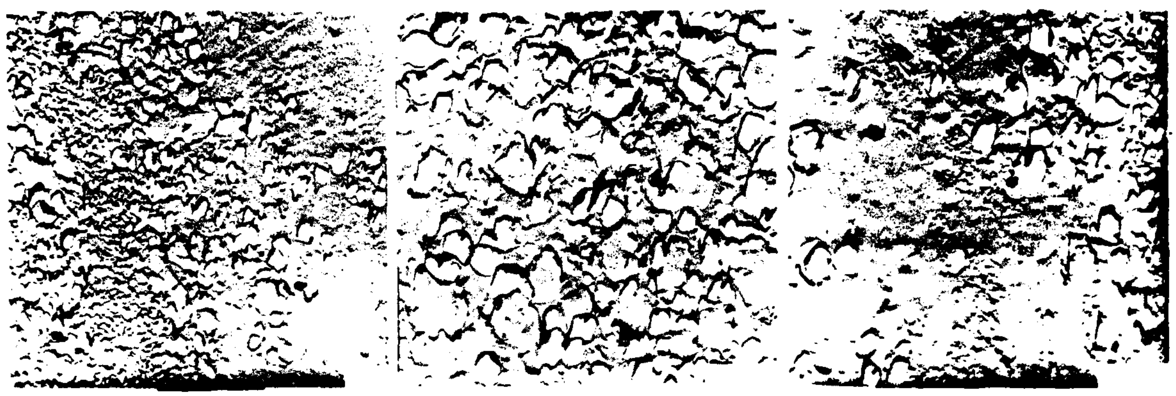
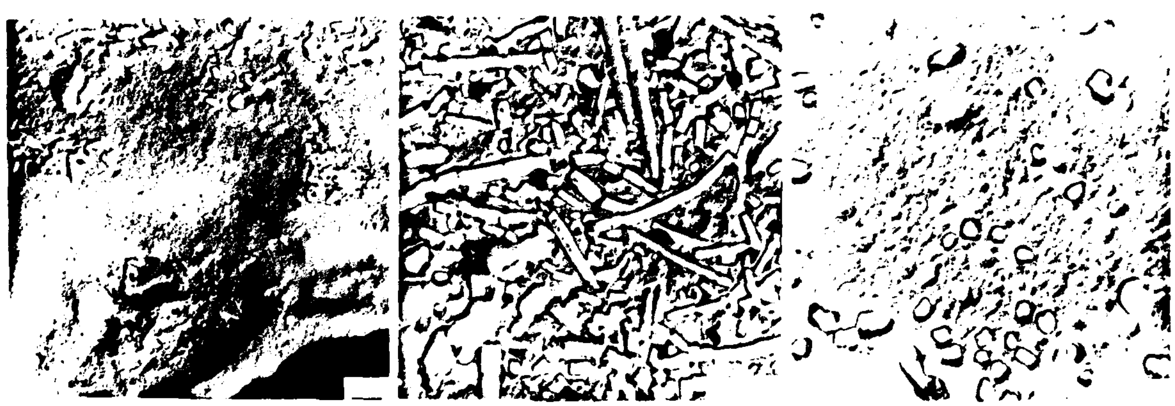


(d) Mag. 5100X (e) Mag. 9600X (f) Mag. 9600X



(g) Mag. 5100X (h) Mag. 9600X (i) Mag. 9600X

Figure 5. Replicas of rolled 2024 aluminum with SiC whiskers as seen in a transmission electron microscope. Specimen was rolled at 840 F at 0.0025 inch per pass. Original thickness was 0.375 inch. Final thickness was 0.020 inch.

from the whiskers. This area is definitely depleted in SiC in contrast to the clumped areas shown previously. Shown in Figure 5d is an inclusion as well as a number of damaged whiskers. Figures 5e and 5f are from longitudinal sections. Figure 5e shows an area of considerable clumping and whisker damage. Intimate contact between neighboring particles occurs. Typical L/D ratios for the fractured whiskers are about 2 to 3. Apparently, rolling in a direction normal to extrusion directions forces the whiskers to bend, thereby inducing fracture. Figure 5f shows another area of sparse SiC content. A number of particles are present but it is not clear whether these are partly fractured whiskers or just SiC powder present in the M-8 whisker material. Figure 5g is a cross-sectional view of the sheet. Clumping effects are again apparent as well as the presence of a lot of very fine particles due to "grinding" of the SiC as a result of the rolling process. In Figures 5h and 5i, cross-sectional views, the most notable feature is the rounding of the hexagonal crystals and the poor dispersion of SiC.

To summarize these results, it can be said that rolling (at least under the conditions experienced by this sheet) imparts very extensive damage into the whiskers. Typical L/D ratios appear to be in the range of 2 to 3 and at that point, the sheet becomes more of a dispersion-strengthened material rather than whisker-reinforced. In addition, the rolling does not appear to alleviate any inhomogeneities in the whisker dispersion which may be present in the as-pressed material or extruded bar.

CONCLUSIONS

Based on the values of mechanical properties measured and reported here, aluminum alloys containing 20 to 25 v/o of M-8 SiC (of which only about 20% is actually whiskers) offer considerable potential for the production of commercially available "super-aluminums." Both high strength and high modulus composite materials are feasible.

In this study, the as-pressed material, the extruded bar, and the rolled sheet represent early developmental attempts to mechanically work and fabricate SiC whisker-reinforced aluminum alloys into useful shapes. The results of the mechanical working strongly indicate that better control of the whisker dispersion and mechanical working conditions is necessary. Porosity and stress concentrators arising from clumping, for example, undoubtedly detract from the mechanical properties of the worked materials. Elimination of these defects and closer control of working temperatures and other processing parameters to minimize whisker damage is necessary. It is recommended that a comprehensive study on the effects of processing parameters be carried out in order to determine the optimum secondary working conditions for these composites.

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